1 458 166

PATENT SPECIFICATION

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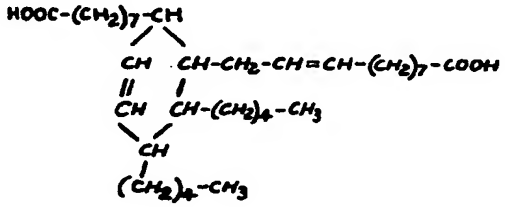
(54) AQUEOUS ANTISEPTIC COMPOSITIONS

(71) We, PENNWALT CORPORATION, a corporation organised and existing under the laws of the State of Pennsylvania, United States of America, of Pennwalt Building, Three Parkway, Philadelphia, Pennsylvania 19102, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

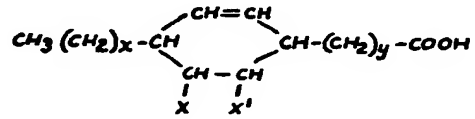
U.S. 1,953,413 discloses an antiseptic preparation containing *p*-chloro-sym. -*m*-xylenol (PCMX) incorporated in an aqueous vehicle, employing a soap as a dispersing agent, such as the soluble alkali metal soap of fatty acids of corn oil, linseed oil, olive oil, castor oil, or other vegetable or animal fatty oils. It is further taught in the sales brochure of the Ottawa Chemical Co., Toledo, Ohio, that alcoholic solutions of *p*-chloro-*m*-xylenol (sold under the trademark "Ottasept" as an antimicrobial additive) are soluble in liquid soaps such as the potassium and triethanolamine salts of oleic, ricinoleic, myristic, and coconut fatty acids. U.S. 3,326,808 concerns a liquid antiseptic detergent composition containing at least one disinfectant agent selected from the group consisting of hexachlorophene, fluorophene, *p*-chloro-*m*-xylenol, bithionol, biphenamine hydrochloride, and a synergistic mixture consisting essentially of 20% of 5,4'-dibromosalicylanilide and 80% of 3,5,4'-tribromosalicylanilide, a water-soluble, non-irritating and non-sensitizing surface-active, organic synthetic anionic detergent; a super fatting emollient selected from the group consisting of animal, vegetable and mineral oils and synthetic fatty acid ester oils, and a polyethylene glycol ether of a higher fatty alcohol. (It is noted that the most widely used germicidal liquid hand soaps heretofore have contained hexachlorophene or iodine; however, hexachlorophene is now out of favor because of recent disclosures relating to possible toxicity, and iodine is undesirable because of its staining properties.) Other aqueous soap-containing detergent compositions containing chloro-xylenols are taught in U.S. 2,191,405, 2,906,664 and 3,370,014. U.S. 3,538,009 concerns an aqueous detergent composition, which may contain a bacteriostatic agent, and which contains a mildness additive comprising the polymerized product of 2 to 4 molecules of a monomeric C_{12} to C_{26} fatty acid, preferably the dimer acid derived from linoleic acid, said dimer acid having the structure



I

However, it has been discovered that soaps of such acids, in common with soaps of many fatty acids derived from naturally occurring vegetable and animal fats, such as ricinoleic and coconut fatty acids, do not operate to give suitable bactericidal and germicidal compositions in combination with *p*-halo-*m*-xylenol (PHMX) antimicrobial agents.

The present invention is based on the discovery that useful aqueous antiseptic compositions comprising a *p*-halo-*m*-xylenol as an antimicrobial agent can be obtained using as the detergent component a water-soluble derivative of a dicarboxylic acid of the formula



II

where x and y are each integers of from 3 to 9 and together total 12 and where one of X and X' is hydrogen and the other is —COOH, the particular derivatives used in accordance with this invention being alkali metal, ammonium and amine salts of the acid itself and of the mono- or di- (2'-hydroxy-3'-sulfo-*n*-propyl) ester of the acid. This diacid is sold by Westvaco Corp. under the mark "Westvaco DiAcid 1550", and the potassium and sodium soaps thereof show remarkable water solubility in that liquid soap solutions up to 80% solids (K soap) or 65% solids (Na soap) can be prepared. The preferred diacid is the reaction product of the linoleic acid portion of a fatty acid mixture with acrylic acid as taught in U.S. Patent No. 3,753,968. Preparation of the mono- and bis-hydroxy propane sulfonate esters of the diacid is taught in U.S. patent No. 3,842,119. The diacid and certain salts thereof are also disclosed in U.S. Patent No. 3,734,859.

In accordance with the present invention, therefore, there is provided an aqueous antiseptic composition comprising: i) from 30—90% water, ii) 2—10% of a *p*-halo-*m*-xylenol and iii) 5—50% of a water-soluble alkali metal, ammonium or amine salt of (a) an acid of Formula II as defined above, or (b) a mono- or di-(2'-hydroxy-3'-sulfo-*n*-propyl) ester of such an acid; such percentages being by weight of the composition, and with the proviso that the amount of component iii) does not exceed its solubility limit in the composition.

When using the water-soluble alkali metal, e.g. sodium, potassium and lithium, or ammonium or amine salts of the dicarboxylic acid of formula II, hereinafter called "the diacid", either the mono- or di-salts may be used.

The preferred detergent components are derivatives of a diacid of Formula II where x is 5, y is 7, X is —COOH and X' is hydrogen.

The preferred aqueous antiseptic compositions of this invention contain from 8 to 18% of the diacid salt or ester, from 2 to 5% of PHMX, preferably p-chloro-*m*-xylenol and from 60 to 80% of water. The potassium and amine soaps wherein the diacid is saponified to an extent of from 50% to 75% are preferred since such compositions are less basic and more soluble than soaps containing other cations; the triethanolamine soaps are of particular value because they are less irritating to the skin. Especially of interest are compositions containing at least 3.75% by weight of *p*-chloro-*m*-xylenol, at least 10% by weight of a sodium or potassium or triethanolamine salt of a diacid of Formula II where x is 5 and y is 7 and optionally up to 20% by weight of an alcohol as a solubilizer for the PCMX.

The compositions of the present invention may be prepared simply by dissolving in water appropriate amounts of PHMX and the diacid salt or ester to provide ingredient concentrations within the expressed ranges. At the lower concentrations of PHMX (i.e. below about 2% by weight) the composition is less bactericidal (i.e. at times only partial kills are experienced) while compositions containing more than 2% (preferably at least 3.75%) by weight of PHMX are completely bactericidal (i.e. 100% kill). Although PHMX is only sparsely soluble in water its water solubility is enhanced by the presence of the diacid derivative. In addition it has been found that the presence of minor amounts (i.e. 1—5%) of an alcohol or glycol supplementary solubilizer will aid the solubility of the PHMX and improve the stability of the final composition during storage.

During the mixing operation it has been found essential to add the ingredients to the water. Reversal of the recommended order of addition, i.e. adding the water last, has been observed to result in compositions ineffective as bactericides presumably due to micellization that masks the PHMX. The preferred order of

addition of ingredients is first to mix the PHMX, the diacid or ester, which may be in the free acid or salt form, and solubilizer (where used) and to add this mixture to the required amount of water containing a chelating agent as may be appropriate, the water also containing, in the case that the free acid is used, at least part of the alkali metal, ammonium or amine base required to neutralize the acid, the remainder of the base being added subsequently together with the optional ingredients such as surface active agents, emollients and perfumes. A final pH adjustment is made by addition of more alkali or diacid to provide a preferred pH in the range 6 to 10. Alternatively, the PHMX, the solubilizer, if used, and the diacid or ester may be added sequentially to water containing the chelating agent and an initial amount of alkali.

As noted, the presence of the diacid soap identified above, i.e. the specified salt of the diacid or of its specified ester, enhances the solubility of PHMX in water. Thus the amount of PHMX that may be solubilized in any given formulation of the present invention will depend upon the concentration of the diacid soap; it will also depend upon the nature and concentration of any supplementary solubilizer that may be present. Finally it has been observed that the effectiveness of the final composition as a bactericide is generally directly proportional to the solubility of the PHMX in water.

In the accompanying drawing there is shown a plot defining solubility limits and bactericidal effectiveness of related concentrations of *p*-chloro-*m*-xylenol (PCMX) and Westvaco Diacid 1550. In the drawing, percentage of Diacid is plotted as abscissa against the percentage of *p*-chloro-*m*-xylenol (PCMX) as ordinate using compositions that otherwise contain the ingredients of the soap of Example 1. The Example uses isopropyl alcohol at a 5% by weight level as a supplementary solubilizer. Curve "A" represents the solubility limit of the PCMX in the system; above curve A, the PCMX component crystallizes out. Curve C represents the dividing line between complete bactericidal versus partial bactericidal activity, i.e. concentrations within the area between curves A and C represent concentrations that are 100% bactericidal in nature whereas those below C are generally less than 100% bactericidal. Concentrations of diacid soap and PCMX between curves C and B represent preferred concentrations not only because of high bactericidal activity but also because of the high shelf-life of the resulting compositions.

It will be obvious to those skilled in the art that the curves will depend upon the identity of the critical components and the supplementary solubilizer. However from this teaching those skilled in the art can readily determine the most effective proportion of ingredients of any combination.

The use of small amounts of alcohols and glycols as supplementary solubilizers for PHMX has been discussed above. While up to 20% by weight of supplementary solubilizer will be found effective in making compositions of the present invention, usually the minimum amount will be employed not only because of safety (flash-point) and cost considerations but because such substances tend to defat the skin, causing dryness. Thus the use of from 1 to 5% by weight of supplementary solubilizer is preferred. Among suitable supplementary solubilizers are alcohols, glycols and sulfonated fatty acids such as hexylene glycol, ethylene glycol, sulfonated oleic acid and the like.

In addition to increasing the solubility of the PHMX in water, the presence of the solubilizer appears to enhance the antimicrobial properties of the PHMX so that a concentration of PCMX as low as about 2% by weight has been found effective to provide bactericidal protection (equivalent to the action of 50 ppm available chlorine) whereas in the absence of the solubilizer about 3.75% by weight of antimicrobial is required to reach such a level of effectiveness.

There are a number of ingredients which may be optionally included in the antiseptic compositions of this invention, some of which enhance performance and others of which are cosmetic in nature. Suitable ingredients for use as skin conditioning agents, surface-active agents, organic chelating agents, perfumes and thickening agents are well known to those skilled in the art. Thus from about 1 to about 5% by weight of a polyhydric alcohol such as glycerol or sorbitol is useful as an additive to provide skin conditioning or emollient properties. As a surface-active agent the composition of present invention will usually have incorporated therein from about 0.5 to about 15% by weight (preferably from 1 to 3% by weight) of an amphoteric or anionic surface-active agent, for example, alkyl imidazolinium dicarboxylate sodium salt, alkyl benzene sulfonate, alkyl sulfate, alkyl benzene sulfate, alkyl sulfonate, alkyl ether sulfonate or like anionic sulfonated detergent;

higher concentrations, e.g. 10% by weight, are often useful when high foam generation is desired. A particularly preferred detergent is a sodium salt of a lauryl sulfate of an alkylphenoxypoly (ethyleneoxy) ethanol made from naturally occurring lauric acid.

For some applications, the use of from about 0.05 to about 5% by weight of an organic chelating agent to enhance the biological activity by sequestering water hardness or other metal ions from the system is beneficial. Representative chelating agents are the alkali metal salts of ethylene diaminetetraacetic acid, the sodium salt of hydroxyethylene diamine triacetic acid, 1-hydroxy-ethylidene-1, 1-diphosphonic acid, nitrile triacetic acid, sodium salts of fluconates, hepta gluconates and citrates. Conventional water soluble perfumes may also be desirably added to enhance the attractiveness of the germicidal hand soap, for example, in amounts ranging from about 0 to 2% by weight.

Thickeners may be used to adjust the viscosity of the final product. Carboxymethylcellulose, carboxyethylene ether cellulose and the like are suitable and may be added in sufficient quantity to provide a wide range of products from a liquid to a thick paste.

Supplementary antimicrobial agents can also be added to the composition. While such additions have been found useful in reducing the amount of PHMX necessary to provide antimicrobial activity, none has been observed to make the PHMX bactericidal below a concentration of PHMX of 2% by weight. Suitable supplementary antimicrobials are 2,4,4'-trichloro-2'-hydroxy-diphenyl ether (Irgasan DP300), "Troysan 142" (made by the Troy Chemicals Co.) and "Santophen* 1" (made by the Monsanto Corp.)

Since the compositions of this invention are primarily intended for topical application to the skin, a pH adjustment close to neutral or slightly alkaline is recommended. Generally this is done by addition of aqueous alkali (such as potassium hydroxide) to the final composition to provide a pH in the range 6 to 10.5. While a higher pH can be used, the increased alkalinity tends to reduce the antimicrobial activity and to increase irritability of the composition upon application to the skin.

In the Examples that follow the bactericidal efficacies of the various compositions are determined using the standard A.O.A.C. test as set forth in Methods of Analysis, 11th Edition (1970) pg. 65, that is, the Available Chlorine Germicidal Equivalent Concentration Tests against the test organisms *Staphylococcus aureus* (ATCC 6538) and *Salmonella typhosa* (ATCC 6539).

Examples

The following Examples are intended to illustrate the invention. They are not intended to limit it in any manner.

Example 1.

The following ingredients are mixed together in a mixing vessel with agitation:

Ingredients	% by Weight
Water	63.95
EDTA (1)	0.10
Natrosol* 250 HR (2)	0.20
Potassium Hydroxide (45% solution)	5.00
Ottasept Technical (3)	3.75
Isopropyl Alcohol	5.00
Westvaco Diacid 1550 (4)	10.00
Sodium Lauryl Sulfate (30%) (5)	10.00
Glycerol	2.00

*Registered Trade Mark.

Notes to table I

(1) Sodium salt of ethylenediaminetetraacetic acid
(2) Hydroxyethylene ether of cellulose
(3) Para-chloro-meta-xylenol
(4) 5-Carboxy-4-hexyl-2-cyclohexene-1-octanoic acid
(5) Alipal Co 433 (Ciba-Geigy) from naturally occurring lauric acid.

In mixing the above ingredients, the chelating agent (EDTA) is first added to the water followed by the alkali. Sufficient alkali is used to form slightly in excess of that required to form the half soap. Thereafter the bactericide (*p*-chloro-*m*-xylenol) is stirred in followed by the solubilizer (isopropyl alcohol) and the diacid (Westvaco Diacid) to form a stable complex. Then the surface active agents, builders, emollients and perfumes and the like are added as desired.

The composition is tested on *Salmonella typhosa* (ATCC No. 6539) using Letheen Broth. Sodium hypochlorite is used as a control. The results where "+" indicates growth and "–" indicates absence of growth, are given below:

Example I

Germicide	Subculture Series									
	1	2	3	4	5	6	7	8	9	10
Sample A	–	–	+	–	–	–	–	–	–	–
Sample B	–	–	–	–	–	–	–	–	–	–

Control (NaOCl)	Conc. ppm Avail. Cl	Subculture Series									
		1	2	3	4	5	6	7	8	9	10
	200	–	–	–	–	–	+	+	+	+	+
	100	–	–	–	+	+	+	+	+	+	+
	50	–	–	+	+	+	+	+	+	+	+

Comparative Control

Culture Resistance to Phenol

Dilution	Intervals in Minutes		
	5 min.	10 min.	15 min.
1:80	+	–	–
1:90	+	+	–
1:100	+	+	+

From the above it will be noted that the composition of Example I is more effective as a bactericide against *Salmonella typhosa* than chlorine at a concentration of 200 ppm where the culture resistance to phenol is 1:80.

The composition of this Example 1 is further tested on *Staphylococcus aureus* (ATCC No. 6538) with the following results

Example I

Germicide	Subculture Series									
	1	2	3	4	5	6	7	8	9	10
Sample C	–	–	–	–	–	–	+	–	–	–
Sample D	–	–	–	+	–	–	–	–	–	–
Sample E	–	–	–	–	+	–	–	+	–	–
Sample F	–	–	–	–	–	–	–	–	–	–

Control	Concn. ppm	Subculture Series									
NaOCl	Avail. Cl	1	2	3	4	5	6	7	8	9	10
	200	–	–	–	–	–	+	+	+	+	+
	100	–	–	–	+	+	+	+	+	+	+
	50	–	–	+	+	+	+	+	+	+	+

Comparative Control

Culture Resistance to Phenol

Dilution	Intervals in Minutes		
	5 min.	10 min.	15 min.
1:60	–	–	–
1:70	+	–	–
1:80	+	+	+

From the above it will be noted that the composition of Example 1 is more effective as a bactericide against *Staphylococcus aureus* than chlorine at a concentration of 200 ppm where the culture resistance to phenol is 1:70.

While a technical grade of the PCMX was used in this Example, a more pure, recrystallized form is available and may be used. The recrystallized form is preferred in applications where a low odor level is important.

The compositions of the present invention are useful as a surgical hand scrub, an anti-microbial soap, as a skin antiseptic or as a health care personnel hand wash. Among other locations the present composition has been recommended for use in official establishments operating under the Federal meat and poultry products inspection program. In such use, the composition is dispensed from adequate dispensers located a sufficient distance from the processing line to preclude accidental product contamination. The hands need not be washed prior to the use of the composition, but afterwards must be thoroughly rinsed with potable water. The composition as it is proposed for use in official establishments is free rinsing and, will not deleteriously affect the meat and/or poultry being processed.

In Examples 2 to 14 presented below in Table 1, various compositions within the scope of the invention are illustrated. Each is effective as a bactericide. In each of the Examples of Table 1 except Example 11, the dicarboxylic acid is Diacid 1550; in Example 11, the dicarboxylic acid is the bis-hydroxypropane sulfonate of Diacid 1550. The letters "N. M." in the Table indicates that the value was not measured. In Example 14 there are additional ingredients not listed in the Table as follows: 2.0% by weight of 2,4,4-trichloro-2-hydroxy-diphenyl ether (supplementary antimicrobial) and 5.0% by weight of propylene glycol (as supplementary solubilizer).

TABLE 1

Example	2	3	4	5	6	7	8	9	10
Water	66.0	59.3	61.2	61.75	61.50	61.25	62.0	61.0	68.2
Dicarboxylic Acid	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0
PCMX	5.0	5.0	5.0	4.25	4.50	4.75	4.0	5.0	5.0
Potassium hydroxide (45% aqueous solution)	8.7	—	8.7	8.7	8.7	8.7	8.7	8.7	8.7
Triethanolamine (98% aqueous solution)	—	10.0	—	—	—	—	—	—	—
Sodium lauryl sulfate (30% aqueous solution)	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	—
Isopropyl alcohol	—	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Glycerol	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	—
Ethylene diaminetetracetic acid, sodium salt	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Water-soluble perfume	0.2	—	—	0.2	0.2	0.2	0.2	0.2	—
pH of composition	10.5	7.5	10.8	N. M.	N. M.	N. M.	10.2	9.5	10.2

TABLE 1 (continued)

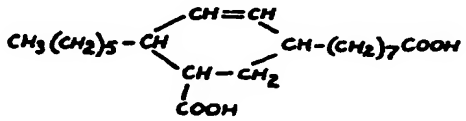
Example	11	12	13	14
Water	63.2	65.15	62.25	57.6
Dicarboxylic Acid	15.5	10.0	13.0	13.0
PCMX	5.0	3.75	3.75	2.0
Potassium hydroxide (45% aqueous solution)	4.0	—	8.7	8.3
Triethanolamine (98% aqueous solution)	—	9.0	—	—
Sodium lauryl sulfate (30% aqueous solution)	5.0	5.0	5.0	5.0
Isopropyl alcohol	5.0	5.0	5.0	5.0
Glycerol	2.0	2.0	2.0	2.0
Ethylene diaminetetracetic acid, sodium salt	0.1	0.1	0.1	0.1
Water-soluble perfume	0.2	—	0.2	—
pH of composition	10.5	N. M.	N. M.	

Example 15.

While PCMX is the preferred microbial for use in compositions of the present invention, other para-halogenated meta-xylenols have been found effective. Thus in tests using parabromo-m-xylenol as the microbial against *Salmonella typhus*, three out of ten bottles displayed an effective kill, equivalent to available chlorine at a 100 ppm concentration.

In Table 2, a series of Comparative Control compositions are listed which are presented to illustrate the critical nature of the antimicrobial and dicarboxylic acid combination and solubility requirements of the present invention. In each of formulations D, E, F and G the solubility limit of the diacid salt was exceeded and the diacid salt crystallized out. None of the compositions of Table 2 is an effective bactericide despite the close similarity of the formulations to those of Table 1. In Table 2 the various superscripts ("a" through "n") are used to identify the carboxylic acid and antimicrobial employed. these are identified as follows:

(a): ricinoleic acid (12-hydroxyoleic acid)
(b): para-chloro-m-xylenol
(c): a mixture of 75% dimer acid (C_{36} dibasic acid) and 22% trimer acid (C_{54} tribasic acid) with 3% monobasic acid (C_{18} fatty acids), sold as "Empol 1022" by Emery Industries, described in U.S. Patent No. 3,538,009.
(d): Westvaco Diacid, 1550



(e): polyethylene glycol ester of above dicarboxylic acid (d)
(f): 1-(3-chloroalkyl)-3,5,7-triaza-1-azoniaadamantane chloride, sold as "Dowicide* 100" germicide by Dow Chemical Co.
(g): an isomer of 1-(3-chloroalkyl)-3,5,7-triaza-1-azoniaadamantane chloride, sold as "Dowicide* 200" germicide by Dow Chemical Co.

*Registered Trade Marks.

(h): aqueous solution of an amine and 1,2-benzisothiazolin-3-one, sold as "Proxel* CRL" germicide by ICI America, Ltd.
(i): sodium salt of 2-mercaptopyridine-N-oxide, sold as sodium omidine germicide by Olin Corp.
(j): tris(hydroxymethyl) nitromethane, sold as Tris Nitro germicide by Commercial Solvents Corp.
(k): 3,5-dimethyltetrahydro-1,3,5-2H-thiadiazine-2-thione, sold as "Troysan 142" by Troy Chemicals Co.
(m): ortho-benzyl-para-chlorophenol, sold as 75% solution in isopropanol under the mark "Santophen* 1" by Monsanto Corp.
(n): distilled coconut acid, a blend of 8% caprylic, 7% capric, 48% lauric, 18% myristic; 9% palmitic, 2% stearic, 8% oleic and 1% linoleic; sold as "Emery 621" by Emery Industries.

TABLE 2

Comparative Controls	A	B	C	D	E	F	G	H	I
Water	62.2	65.9	64.7	63.1	66.4	63.6	66.9	65.2	65.5
Carboxylic Acid	16.0[a]	15.9[c]	12.0[d]	13.0[d]	10.0[d]	13.0[d]	10.0[d]	15.0[e]	13.0[d]
Antimicrobial	5.0[b]	5.0[b]	5.0[b]	3.5[b]	3.5[b]	3.0[b]	3.0[b]	5.0[b]	0.5[f]
Potassium hydroxide (45% aqueous solution)	4.5	8.1	—	8.3	—	8.3	—	—	8.7
Triethanolamine (98% aqueous solution)	—	—	6.0	—	8.0	—	8.0	2.5	—
Sodium lauryl sulfate (30% aqueous solution)	5.0	—	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Isopropanol	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Glycerol	2.0	—	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Ethylene diaminetetracetic acid, sodium salt	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Perfume	0.2	—	0.2	—	—	—	—	0.2	0.2
pH	9.5	11.2	7.8	10.1	7.7	10.3	7.6	N. M.	N. M.

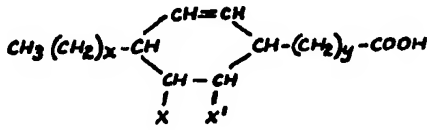
* Registered Trade Marks.

TABLE 2 (continued)

Comparative Controls	J	K	L	M	N	O	P	Q	R
Water	65.0	65.5	65.7	65.5	65.75	64.3	61.7	69.7	66.4
Carboxylic Acid	13.0[d]	13.0[d]	13.0[d]	13.0[d]	13.0[d]	13.0[d]	13.5[n]	13.5[n]	13.5[n]
Antimicrobial	1.0[g]	0.5[h]	0.3[i]	0.5[j]	0.25[k]	6.7[m]	5.0[b]	2.0[g]	0.3[i]
Potassium hydroxide (45% aqueous solution)	8.7	8.7	8.7	8.7	8.7	8.7	7.5	7.5	7.5
Triethanolamine (98% aqueous solution)	—	—	—	—	—	—	—	—	—
Sodium lauryl sulfate (30% aqueous solution)	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Isopropanol	5.0	5.0	5.0	5.0	5.0	—	5.0	—	5.0
Glycerol	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Ethylene diaminetetracetic acid, sodium salt	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Perfume	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
pH	N. M.	N. M.	N. M.	N. M.	N. M.	N. M.	N. M.	N. M.	N. M.

WHAT WE CLAIM IS:—

1. An aqueous antiseptic composition comprising: i) from 30—90% water, ii) 2—10% of a *p*-halo-*m*-xylenol and iii) 5—50% of a water-soluble alkali metal, ammonium or amine salt of a) an acid of the formula:



where x and y are each integers of from 3—9 and together total 12; and one of X and X' is hydrogen and the other COOH, or (b) a mono- or di-(2'-hydroxy-3'-sulfo-

n-propyl) ester of such an acid; such percentages being by weight of the composition, and with the proviso that the amount of component iii) does not exceed its solubility limit in the composition.

2. A composition according to claim 1, wherein component ii) is *p*-chloro-*m*-xylenol.

3. A composition according to claim 1 or 2, which contains from 2—5% by weight of *p*-halo-*m*-xylenol.

4. A composition according to any one of claims 1—3, wherein component iii) is present in an amount of from 8—18% by weight.

5. A composition according to any one of claims 1—4, wherein component iii) is a sodium, lithium, or potassium salt of said acid or ester.

6. A composition according to any one of claims 1—4, wherein component iii) is a triethanolamine salt of said acid or ester.

7. A composition according to any one of claims 1—4, wherein component iii) is a mixture of potassium or amine salts of said acid in which mixture from 50—75% of the carboxylic acid groups are in salt form.

8. A composition according to any one of claims 1—7, which contains from 60—80% by weight of water.

9. A composition according to any one of claims 1—8, which also contains up to 20% by weight, based on the total composition of a solubilizer for the *p*-halo-*m*-xylenol, being a compound other than said salt.

10. A composition according to claim 9, in which the solubilizer is an alcohol, glycol or a sulfonated fatty acid.

11. A composition according to claim 9 or 10, which contains from 1—5% by weight of said solubilizer.

12. A composition according to any one of the preceding claims, which additionally contains one or more of the following: a skin conditioning agent, a surface active agent, an organic chelating agent, a thickener, or a perfume.

13. A composition according to claim 1, substantially as hereinbefore described in any one of the foregoing Examples.

14. A method of making a composition according to claim 1, which comprises mixing the *p*-halo-*m*-xylenol with said salt, or with the corresponding free acid and adding the mixture so formed to water, the water containing, in the case where the free acid is used, at least part of the alkali metal, ammonium or amine based required to neutralize said acid, the remainder of the base being added subsequently.

15. A method according to claim 14, as applied to the preparation of a composition as claimed in any one of claims 2—12.

16. A method according to claim 15, being applied to the preparation of a composition according to claim 9, 10 or 11, wherein the solubilizer is added to the mixture of the *p*-halo-*m*-xylenol and the acid or salt before the addition of the water.

17. A composition according to claim 1 comprising at least 3.75% by weight of *p*-chloro-*m*-xylenol and at least 10% by weight of a sodium, potassium or triethanolamine salt of a dicarboxylic acid of the formula specified where x is 5 and y is 7.

18. A composition according to claim 17, which also contains up to 20% by weight of an alcohol as a solubilizer for the *p*-chloro-*m*-xylenol.

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